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A Survey of Current High-Performance Carbon Fiber Characterization Methods

by Steven P. Nguyen, Linda L. Ghiorse,
and Thomas J. Mulkern

ARL-TR-2293 August 2000

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A Survey of Current High-Performance Carbon Fiber Characterization Methods

Steven P. Nguyen, Linda L. Ghiorse,
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Abstract

This report reviews various test techniques published in the literature for evaluating carbon fiber via the single-filament tensile test, the dry bundle test, the resin-impregnated strand test, and the single-fiber fragmentation test (optical microscopy and acoustic emission). Experimental procedures, data analysis, and statistical tensile strength theory are also described. Each technique is followed by a discussion of the advantages and limitations. Furthermore, a materials property database has been developed that includes mechanical properties for several commercially available carbon fibers.

Acknowledgments

The authors would like to thank Dr. Steven H. McKnight and Seth R. Ghiorse of the U.S. Army Research Laboratory (ARL) for their review and technical comments on this report.

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1. Introduction

This is part one of a two-part review of the physical properties of high-performance, Polyacrylicnitrile (PAN) based carbon fibers, and the techniques used in characterizing those properties. This report reviews the most common test methods used to determine the tensile properties and the statistical strength distribution of fibers—the single-filament tensile test, the tow test (dry bundle and resin-impregnated tow test), and the single-filament fragmentation test (optical microscopy and acoustic emission). This part of the study also contains the updated physical properties of the most popular PAN-based carbon fibers currently available on the market (see the Appendix). The second part of the study will discuss other characterization techniques used in determining the microstructures of the fibers and the relationship between these fine structures and the tensile properties of carbon fibers. The purpose of this review is to provide guidance for testing fibrous reinforcements. In addition, this report will serve as a resource for the properties of selected carbon fiber materials.

2. Test Techniques

2.1 Single-Filament Tensile Test. The first test used in determining tensile properties of carbon fibers is the single-filament tensile test. This test requires carefully extracting an individual carbon fiber from a tow, and great effort goes into aligning the test fiber axially. The test also calls for a significant number of samples for statistical analysis.

The following test procedure follows the ASTM Standard D3379 (American Society for Testing and Materials 1989). Individual fibers are randomly selected from a tow, and each fiber is carefully center-line mounted on a thin paper tab, as shown in Figure 1. Fibers are held in place by adhesive at the edges of slots in the tab. The slot length is the gage length of the test specimen, which is usually between 20 and 30 mm. The tab should be about three times longer than the specimen's gage length and half of the width of the gage length. In a tensile test device, the prepared specimen is then axially gripped at the tab ends. If the direct strain measurement of

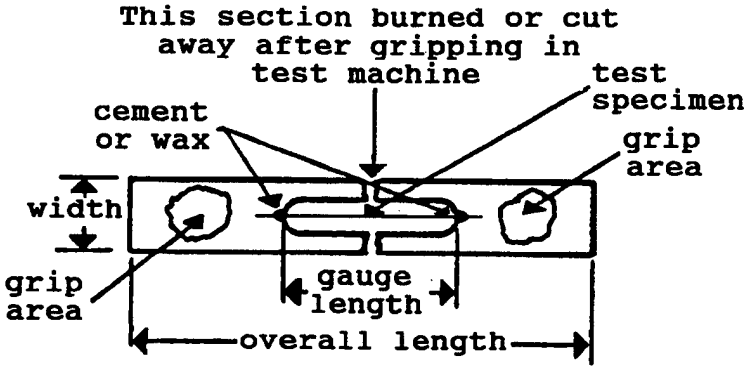

Figure 1. Single-Fiber Mounting Tab.

the specimen gage section is not possible, the compliance of the test device should be determined. The true compliance can then be calculated for determining Young's modulus.

2.1.1 Diameter Measurement. It is necessary to measure fiber diameter to calculate strength and modulus. Several methods are available, such as the optical microscope, the scanning electron microscope (SEM), and laser diffraction. Studies have shown that optical microscopic measurements tend to have a higher standard deviation than other methods (Chen and Diefendorf 1982). SEM methods can produce precise measurements of fiber diameter, but the procedure requires more sample preparation and image manipulation. This technique is also not practical for measuring the diameters of specimens to be tested. Fukuda et al. (1993) found that a laser diffraction method was most suitable for the 1 × 1 diameter measurements of the fibers to be tested. In this technique, fiber diameters are determined by measuring the distance between dark spots of the diffraction pattern, as in Figure 2.

The diameter, d, is calculated as follows:

$$d = \frac{2 L \lambda}{1}, \qquad (1)$$

where L is the distance between the specimen and the screen, λ is the wavelength of the laser light, and 1 is the distance between dark spots.

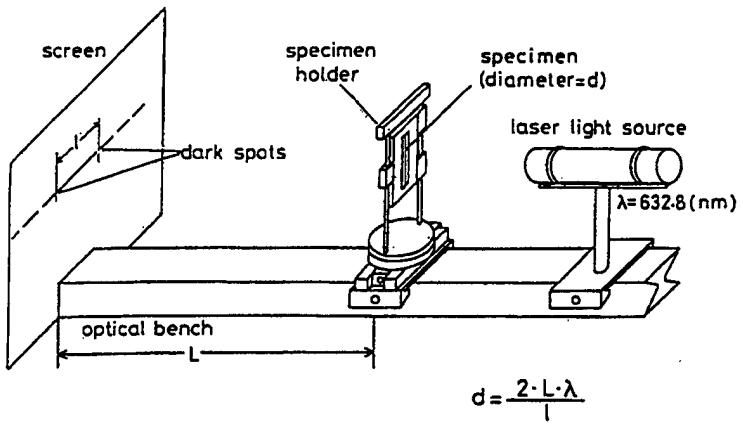

Figure 2. Schematic View of Laser Diffraction Diameter Measurement (Fukuda et al. 1993).

*2.1.2 **Statistical Tensile Strength.*** Unlike metals whose strengths are more deterministic, the strengths of brittle fibers are known to exhibit great variation between individual fibers, as well as along a fiber length. Hence, statistical models are most appropriate to describe strength distributions of fibers, and the best known model in composite strength theory has been the Weibull distribution (Chou 1992). The Weibull distribution of fiber strength, σ_f, has the following form:

$$P_f(\sigma_f) = 1 - \exp\left[-L(\sigma_f/\sigma_0)^{\beta}\right], \tag{2}$$

where P_f is the cumulative probability of failure of a fiber of length L at a stress level $\geq \sigma_f$. The Weibull scale parameter is σ_0, and β is the Weibull shape parameter reflecting the scattering of strength data. Based on experimental data, values of strength parameters σ_0 and β can be determined by various approaches (Manders and Chou 1983). One of these approaches is discussed in the next section. The mean fiber strength can then be calculated by the equation

$$\overline{\sigma} = \sigma_0 L^{-1/\beta}\,\Gamma(1 + 1/\beta), \tag{3}$$

where Γ denotes the gamma function.

2.1.3 Strength of Fibers at Short Lengths. The strengths of fibers also strongly depend on fiber lengths, as shown in equation 3. Often, knowing fiber strengths at very short lengths is crucial, especially in analyzing the fiber-matrix interfacial strength in composites. Testing very short fibers, however, is not feasible with the single-filament tensile test, due to problems with specimen gripping and alignment. Many attempts have been made to extrapolate strengths of short fibers from longer specimens with limited success. Most of these extrapolation methods rely on the assumption that Weibull parameters σ_0 and β are length independent. However, Asloun et al. (1989) found through their experimental data that Weilbull parameters varied with fiber length. Their study also showed that for extrapolating fiber strengths at short lengths, the most accurate and simple method was by means of the linear logarithmic dependence of strength on gage length. By taking the logarithm on both sides, equation 3 becomes

$$Ln\left(\bar{\sigma}_f\right) = (-1/\beta)\ln L + \ln\left[\sigma_0\,\Gamma\left(1 + 1/\beta\right)\right]. \tag{4}$$

The plot of mean fiber strengths vs. gage lengths in a logarithmic scale produces a straight line (Figure 3), which could be extended to estimate fiber strengths at short lengths. The slope of the line is the value of $(-1/\beta)$, and the value of σ_0 is derived from the y intercept. Mean fiber tensile strengths can simply be averaged from the experimental sample size of about 20. It can also be estimated if values of σ_0 and β are known. To achieve good extrapolation results, fiber tensile tests should be performed at many different gage lengths (at least seven), and there should be a sample size of about 20 for each gage length (Asloun et al. 1989). The validity of this method was confirmed in previously published data (Manders and Chou 1983; Goggin 1975; Barry 1978; Hitchon and Phillips 1979; Jones et al. 1980).

2.1.4 Discussion. Although the single-filament tensile test method can provide good tensile properties data and requires simple analysis, it has several disadvantages. The method is very tedious and labor intensive. A large sample size of about 20 is also required for a good statistical analysis. Because it is not always possible to directly measure fiber elongation, the compliance of tensile test devices should be determined. The experimental results could also be

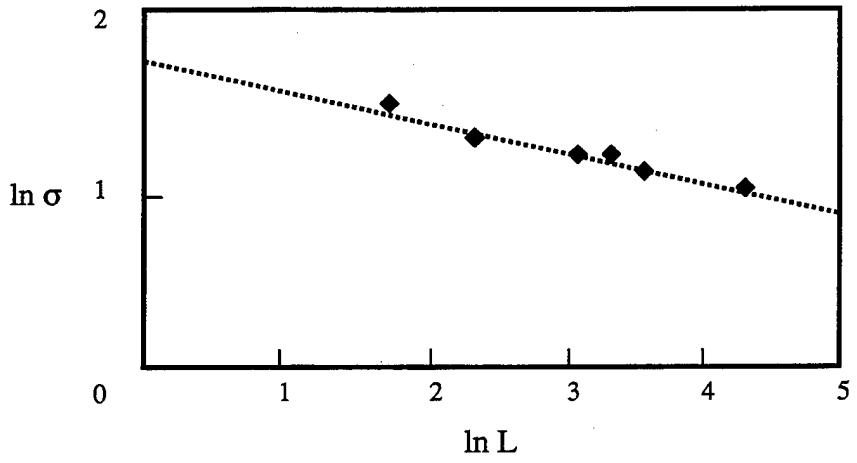

Figure 3. Mean Tensile Fiber Strength vs. Gage Length in Log Scale.

overestimated since only the strong fibers are extracted for tensile tests. Weak fibers break prior to testing and are discarded during sample preparation. In addition, a tensile test of very short fibers (<1 mm) is almost impossible. As a result, extrapolation techniques must be used, and they are usually unreliable. Another test method, the single-fiber fragmentation (discussed in section 2.3), may resolve many of these issues.

2.2 Fiber Tow Test.

2.2.1 Dry Bundle (Tow) Test. Because of the high degree of scattering in strengths of brittle carbon fibers, a large number of specimens are needed for good statistical analysis. With the fiber dry bundle test, this problem is minimized. Since the failure of each fiber bundle is the result of independent fractures of many fibers, there is much less data scattering for single fibers. Therefore, a smaller number of tests is required to achieve statistically valid data. Experimental data indicated that Weibull strength parameters determined from dry bundle tests are very similar to single-fiber tests (Manders and Chou 1983).

The following steps are typical experimental procedures. Fiber tows of different lengths (usually between 5 and 10 tows for each gage length) are cemented into grooved end tabs. There

should be no slack in any of the fibers. The cross-sectional area of the tow can be determined from the manufacturer's data of density and mass per unit length. All fibers are assumed to have the same diameter. The tows are then tested using a tensile test device. Tensile properties are determined from load and strain data similar to the procedure outlined in ASTM Standard D4018 (American Society of Testing and Materials 1993). The statistical analysis of a dry bundle test is similar to that discussed in section 2.1.3. The mean strengths of fiber tows (at different lengths) are plotted against corresponding lengths in logarithmic scale. The plot should approximate a straight line whose slope is $-1/\beta$. Calculated from the y-intercept is σ_0. Mean fiber strengths can then be estimated from the determined Weibull parameters (equation 3).

*2.2.2 **Resin-Impregnated Tow Test.*** A more common and efficient method for determining tensile properties of carbon fiber is the resin-impregnated tow test. This technique is also better than the single-filament tensile method in the sense that fiber properties are measured in an environment more compatible to real composite parts. Experimental data shows that the fiber Young's moduli determined from the strand test method was closer to that obtained from composite data (Kowalski 1988).

The following procedure follows ASTM Standard D4018. Tows of carbon fibers are dipped into a compatible resin system and then worked over rollers or dies to squeeze out excess resin. The resin content should be between 35% and 60% by weight. The resin should also have greater strain-to-failure than the fibers so that it will not affect the evaluation of fiber-tensile properties. The main role of the resin is to provide enough support for specimen handling and a better, more uniform loading among fibers during the tensile test. A recommended system is a combination of bisphenol F epoxy and diethyltoluene diamine (3.9:1 by weight). Resin-impregnated strands are then cured under slight tension. If tabs are used, the distance between tabs should be 150 mm; otherwise, that would be the distance between the test grips. Tensile properties are calculated from load and strain data.

*2.2.3 **Discussion.*** Tow tests have several distinct advantages over single-filament tests. It is more practical to handle carbon fiber tows than single filaments, and similar experimental results

can be achieved in the case of the dry bundle test. In addition, due to less strength scattering in dry bundles, a smaller sample size is needed for testing. For the resin-impregnated tow test, the ease in specimen handling also makes direct strain measurements possible. Measured tensile properties are closer to those of composites, since fibers are surrounded by a polymer matrix. The trade-off is the deviation from the actual values of fiber-tensile properties. Thus, it is important to select a resin system that is compatible to the particular carbon fiber and greater in strain capability.

2.3 Single-Fiber Fragmentation Test. The single-fiber fragmentation test offers yet another technique for determining the strength of fibers, especially very short fibers, which has always been an obstacle in other methods. Similar to the resin-impregnated tow test, another advantage of the fiber fragmentation test is that it produces actual composite material data. The amount of data generated by the test is also much larger, making it more suitable for statistical analysis.

In a fragmentation test, a single fiber embedded in resin is strained, while the fiber repeatedly fractures into shorter and shorter fragments due to increasing stress. Because there are many fiber breaks in a single test, an abundance of data is produced in one fragmentation test. The fragmentation test is based on the following simple modification of equation 2:

$$P_s(l) = 1 - P_f(\sigma) = \exp\left[-L(\sigma_f/\sigma_0)^{\beta}\right], \tag{5}$$

where $P_s(l)$ is the survival cumulative probability of fibers at different specified lengths, l. Taking the logarithm of both sides,

$$\ln(P_s) = -L(\sigma_f/\sigma_0)^{\beta}. \tag{6}$$

Thus, by knowing the cumulative probability that the fiber fragments are greater than or equal to a specified length, the plot of $\ln(P_s)$ vs. L can be obtained. The plot should be linear, with a gradient equal to $-(\sigma_f/\sigma_0)^{\beta}$ (see Figure 4).

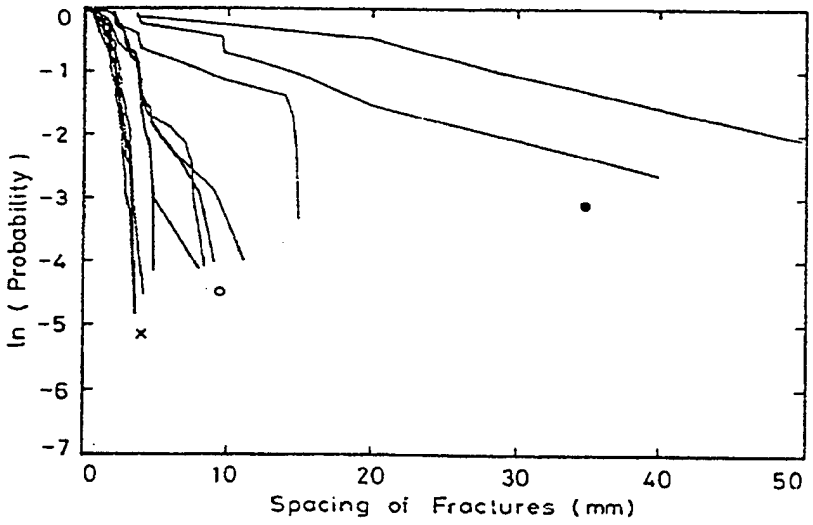

Figure 4. Logarithm of Cumulative Probability That a Fiber Fragment Is Greater or Equal to a Specified Length, $\ln(P_s)$, vs. the Spacing of Fractures, L (Manders and Chou 1983).

These values of gradient are plotted against their corresponding levels of stress or strain (assuming linearly elastic fiber). The plot of ln(gradient) vs. $\ln(\sigma_f)$ or $\ln(\varepsilon_f)$ will then give the slope of value β and the y intercept of value σ_0. Because the embedded fiber in the fragmentation test can fracture into very short segments, the values of β and σ_0 are the actual Weibull parameters of short fibers and are not obtained from extrapolation. Mean fiber strengths are calculated from these strength parameters according to equation 3.

Although there is no standardized procedure for the fragmentation test, various experimental procedures in the literature (Baillie and Bader 1994; Yavin et al. 1991; Gulino and Phoenix 1991; Waterbury and Drzal 1991; Waterbury 1990) follow these essential steps:

(a) A long fiber (longer than 50.8 mm) is carefully selected from a bundle and handled only by its ends. The fiber is then mounted on a silicon rubber mold. The mold is a standard ASTM 50.8-mm long dogbone cavity 3.175-mm wide × 1.587-mm thick × 25.4-mm long at the gage section. The mold cavity has sprue slots at each end to align and center the fiber axially. Once aligned, the fiber is held in place with fast curing epoxy in the sprue slots.

(b) A resin system with greater strain-to-failure than the fiber (at least three times greater) is then either poured or pipetted into the mold cavity. Air bubbles in the resin can be avoided by degassing the mold and resin in a vacuum oven before filling the cavity.

(c) The mold is placed in an oven and the resin is cured with the appropriate cure cycle; the specimen is then removed with great care to avoid damaging the embedded fiber.

(d) The specimen is tested at small strain increments using a microstraining machine.

To determine the cumulative probability, P_s, of fiber fragments in the fragmentation test, the number of fragments and their lengths need to be measured and ranked at each strain level. Currently, there are two different techniques in this data acquisition—optical microscopy and acoustic emission.

2.3.1 Optical Microscopy Data Acquisition. For the optical microscopy technique, the number of fiber fragments and their lengths are measured at each strain level by a traveling microscope that moves along the fractured fiber. Measurements can also be done by mounting the microstraining device on the optical microscope during the translation stage. The optical resolution of the microscope should be powerful enough to identify the microfractures in the fiber. This procedure, however, can take hours to perform. In attempts to improve the speed of data acquisition, Waterbury and Drzal (1991) developed a technique with some automated features that could reduce the data acquisition time by an order of magnitude. Their system includes a computer-interfaced translation stage and a computer program that performs data processing. After each strain increment, the stage is translated at a constant rate while the fractured fiber is observed under the microscope. As fractures pass a set of cross hairs, the operator clicks the mouse and sends timing signals to the computer. The elapsed time between fractures is converted into fragment lengths by the software. Statistical analysis, fiber strength, and interfacial shear strength are also readily performed.

2.3.2 Acoustic Emission Data Acquisition. Despite many advantages over conventional methods, the optical microscopy technique has many shortcomings. First, the selected resin must be transparent so that the embedded fiber can be observed under the light microscope. Second, the strained fiber has to remain strained for a long time during the fragment length measurement. This step can induce creep in the specimen, as shown by Clough and McDonough (1996). Third, it is sometimes very difficult to identify the small gaps between fiber fragments, which are usually <1 μm. The acoustic emission technique could potentially surmount these problems. In this technique, the fiber breaks are determined by detecting the burst of acoustic emission from each fracture. Some variations of this technique monitor acoustic source locations to determine the fragment lengths. Expensive equipment, however, is required. A simpler and more efficient technique was developed by Clough and McDonough of the National Institute of Standards and Technology. Based on the fragmentation theory (Gulino and Phoenix 1991), Clough and McDonough further simplified the analysis under the condition of low-applied fiber stress, σ_f, or applied strain, ε_f (which was measured here). The following equation is derived:

$$N \cong (\sigma_f / \alpha_0)^{\beta}, \tag{7}$$

where N is the number of detected fiber breaks. Thus, the only information needed is the number of breaks and the levels of applied stress or strain. Values of Weibull parameters can be determined from the plot of ln(N) vs. $\ln(\sigma_f)$ or $\ln(\varepsilon_f)$.

To monitor break events, an acoustic emission transducer is attached to the gage section of the specimen. As the specimen is strained at a slow rate (approximately 5×10^{-4} s^{-1}), the emission signals are amplified and recorded continuously by a computer. To avoid yielding, the applied strain should not exceed 2%. After a test, the entire signal recording is postprocessed with a median subtraction filter (Barnett 1989). This particular filter method greatly enhances the trigger signal and is able to filter out noise, but not emission events.

2.3.3 Discussion. The single-fiber fragmentation test is a better method for evaluating fiber strengths at short gage lengths. Experimental data is also more accurate because the

fragmentation test is an in-situ method. The fiber is fractured in a real composite environment. In a single test, a typical 25-mm fiber fractures about 50 times, which is equivalent to 50 single fiber tests (Waterbury and Drzal 1991). The fragmentation test, therefore, is able to generate a much larger amount of test data than other methods. Yet, the test time can be significantly reduced. In the acoustic emission technique by Clough and McDonough (1996), the amount of time required for each test is a minute or less.

Optical microscopy and acoustic emission techniques both produce close experimental results. The optically monitored method, however, requires more human interaction. In addition, the longer test time can cause creep in the specimens, generating error in the data. The acoustic emission technique overcomes the problems of visibility and creep since data is acoustically collected at the precise stress/strain level and not after each stress/strain increment, as in the optical technique (Clough and McDonough 1996). The equipment used for detecting acoustic emission, however, must be very sensitive and capable of filtering out background noise. Despite this, the technique could still be vulnerable to error due to simultaneous, multiple fiber fractures.

3. Conclusion

Each of the test techniques has its own unique advantages and limitations. No single technique is completely superior to others in all aspects. The dry bundle test is relatively better in determining average strengths of fiber and statistical parameters of longer fibers. For more accurate values of Young's modulus, however, the resin-impregnated strand test with an appropriate resin system is better. The single-fiber fragmentation technique is by far the best method for generating a large amount of data for statistical analysis, and it generates better accuracy of fiber strengths at very short lengths.

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Appendix:

High-Performance Carbon Fibers Database

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As a part of this report, a database containing information on selected PAN-based carbon fibers was created using the database software MicroSoft Access. The software allows fast fiber selection by criteria on physical properties such as modulus, strength, density, fiber diameter, and CTE, as well as on manufacturers' information (Figure A-1). If a user wants to select particular fibers of modulus >350 GPa, a simple query can be entered, as shown in Figure A-2. The database then conveniently produces selections quickly (Figure A-3). Also included is updated price information on these high-performance fibers. The mechanical properties of the fibers are provided by the producers of the fibers, and the resin-impregnated strand test is typically used. This database is available from the Polymer Research Branch of the U.S. Army Research Laboratory-Weapons and Materials Research Directorate (ARL-WMRD), Aberdeen Proving Ground, MD.

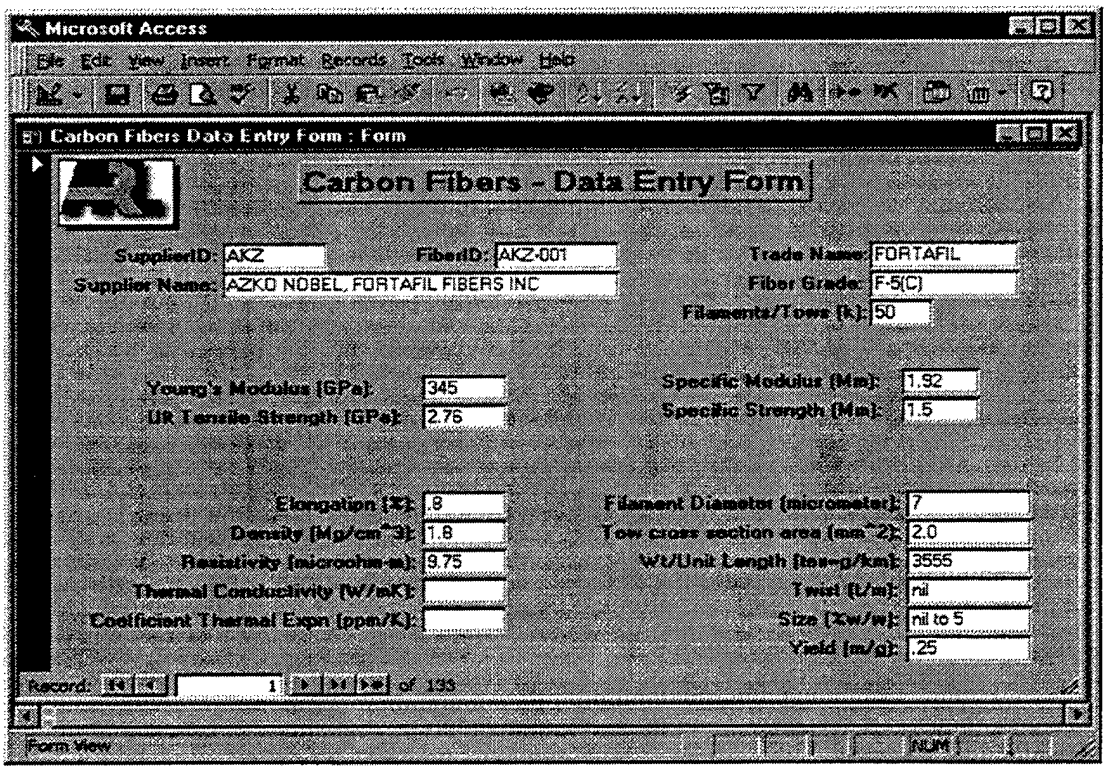


Figure A-1. Data of Selected High-Performance Carbon Fibers in the Database.

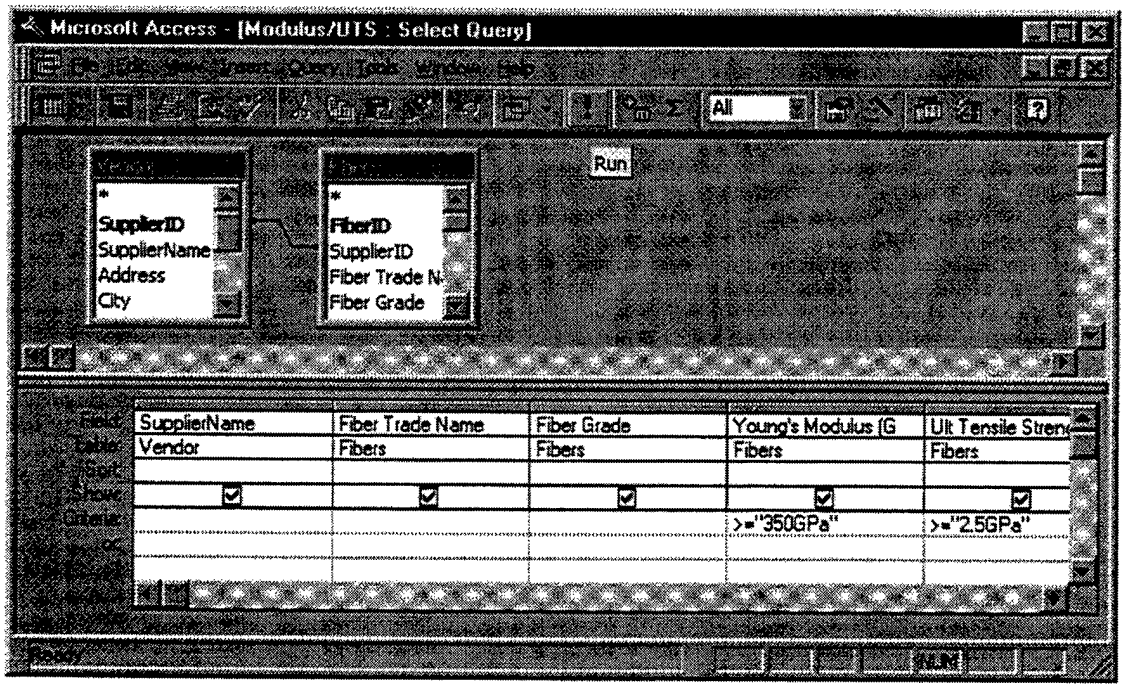
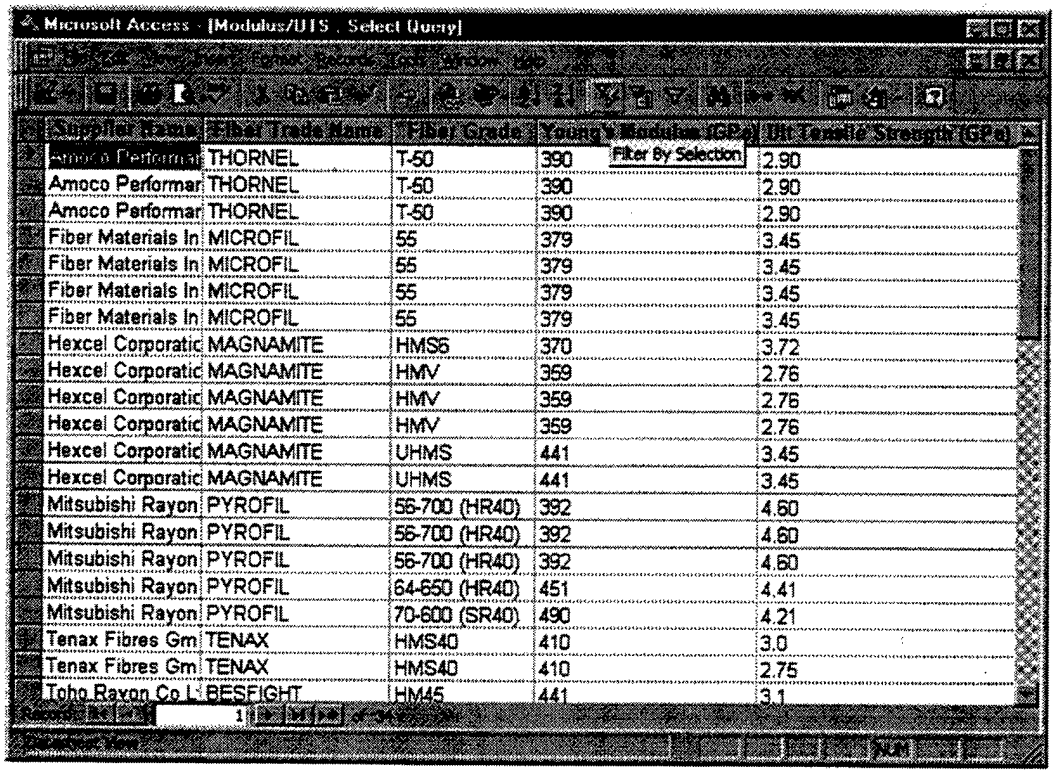


Figure A-2. A Typical Query Format for a Particular Carbon Fiber.



Supplier Name	Fiber Trade Name	Fiber Grade	Young's Modulus (GPa)	Ult Tensile Strength (GPa)
Amoco Performar	THORNEL	T-50	390	2.90
Amoco Performar	THORNEL	T-50	390	2.90
Amoco Performar	THORNEL	T-50	390	2.90
Fiber Materials In	MICROFIL	55	379	3.45
Fiber Materials In	MICROFIL	55	379	3.45
Fiber Materials In	MICROFIL	55	379	3.45
Fiber Materials In	MICROFIL	55	379	3.45
Hexcel Corporatic	MAGNAMITE	HMS6	370	3.72
Hexcel Corporatic	MAGNAMITE	HMV	359	2.76
Hexcel Corporatic	MAGNAMITE	HMV	359	2.76
Hexcel Corporatic	MAGNAMITE	HMV	359	2.76
Hexcel Corporatic	MAGNAMITE	UHMS	441	3.45
Hexcel Corporatic	MAGNAMITE	UHMS	441	3.45
Mitsubishi Rayon	PYROFIL	56-700 (HR40)	392	4.60
Mitsubishi Rayon	PYROFIL	56-700 (HR40)	392	4.60
Mitsubishi Rayon	PYROFIL	56-700 (HR40)	392	4.60
Mitsubishi Rayon	PYROFIL	64-660 (HR40)	451	4.41
Mitsubishi Rayon	PYROFIL	70-600 (SR40)	490	4.21
Tenax Fibres Gm	TENAX	HMS40	410	3.0
Tenax Fibres Gm	TENAX	HMS40	410	2.75
Toho Rayon Co L	BESFIGHT	HM45	441	3.1

Figure A-3. Sorted Results From the Query.

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2	DEFENSE TECHNICAL INFORMATION CENTER DTIC DDA 8725 JOHN J KINGMAN RD STE 0944 FT BELVOIR VA 22060-6218
1	HQDA DAMO FDT 400 ARMY PENTAGON WASHINGTON DC 20310-0460
1	OSD OUSD(A&T)/ODDDR&E(R) R J TREW THE PENTAGON WASHINGTON DC 20301-7100
1	DPTY CG FOR RDA US ARMY MATERIEL CMD AMCRDA 5001 EISENHOWER AVE ALEXANDRIA VA 22333-0001
1	INST FOR ADVNCD TCHNLGY THE UNIV OF TEXAS AT AUSTIN PO BOX 202797 AUSTIN TX 78720-2797
1	DARPA B KASPAR 3701 N FAIRFAX DR ARLINGTON VA 22203-1714
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This report reviews various test techniques published in the literature for evaluating carbon fiber via the single-filament tensile test, the dry bundle test, the resin-impregnated strand test, and the single-fiber fragmentation test (optical microscopy and acoustic emission). Experimental procedures, data analysis, and statistical tensile strength theory are also described. Each technique is followed by a discussion of the advantages and limitations. Furthermore, a materials property database has been developed that includes mechanical properties for several commercially available carbon fibers.

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